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14. ABSTRACT
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Report Title

Final Report: STIR: RDRL-ROE-M: Microwave Response of Carbon Nanotubes in Polymer Nanocomposite Welds

ABSTRACT

Thrust 1 of the STIR project examines the heat response of polymer composites loaded with carbon nanotubes (CNTs) to microwave irradiation. This involves (1) a study of how CNT loading affects dielectric properties of polymer composites and (2) a study of how CNT loading affects the heating response to microwave radiation. Our hypothesis is that the heating of CNTs alone is not the only factor; rather, the formation of resistive (rather than capacitive) percolating CNT networks is the dominant factor in the interaction of the sample with the microwave field and the subsequent heat evolution.

Thrust 2 of the STIR project examines the effects of microwave heating of CNT-based adhesives at welds between polymer films. We hypothesize that localized CNT heating at an interface allows for polymer mobility across the interface can allow the weld to become as strong as the bulk polymer sample. We investigate such welds in both bonded polymer films and printed polymer filament structures.

For our experimental system, we choose polylactic acid (PLA) as a model polymer, given its common application in additive manufacturing. 1-3 For the nanofiller, we utilize multi-walled carbon nanotubes.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received   Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received   Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

SPE Downhole Tools Workshop: Challenges, Opportunities, and Value in HPHT Applications, Galveston, TX, Oct 2015
Presentation: "Microwave Induced Welding of Carbon Nanotube-Thermoplastic Interfaces for Enhanced Mechanical Strength," C.B. Sweeney, B.A. Lackey, M.A. Saed, M.J. Green [Speaker]

AIChE Annual Meeting, Salt Lake City, UT, Nov 2015
Presentation: "Microwave Induced Welding of Carbon Nanotube-Thermoplastic Interfaces," C.B. Sweeney, B.A. Lackey, M.A. Saed, M.J. Green [Speaker]

APS March Meeting, San Antonio, TX, Mar 2015
Presentation: "Microwave induced welding of carbon nanotube-thermoplastic interfaces for enhanced mechanical strength of 3D printed parts," C.B. Sweeney [speaker], M.J. Green, M.A. Saed

Number of Presentations: 3.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u>   <u>Paper</u>

01/28/2016 3.00 Sweeney, C.B., Saed, M.A., Green, M.J.. Microwave induced welding of carbon nanotube-thermoplastic interfaces for enhanced mechanical strength of 3D printed parts,
APS March Meeting. 04-MAR-15, . : ,

01/28/2016 2.00 Sweeney, C.B., Saed, M.A., Green, M.J.. Microwave Induced Welding of Carbon Nanotube-Thermoplastic Interfaces,
AIChE Annual Meeting. 10-NOV-15, . : ,

01/28/2016 4.00 Sweeney, C.B., Lackey, B.A., Saed, M.A., Green, M.J. Microwave Induced Welding of Carbon Nanotube-Thermoplastic Interfaces for Enhanced Mechanical Strength,
5. SPE Downhole Tools Workshop: Challenges, Opportunities, and Value in HPHT Applications. 20-OCT-15, . : ,

    TOTAL: **3**

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

<u>Received</u>   <u>Paper</u>

01/28/2016   1.00   Sweeney, C.B., Saed, M.A., Green, M.J.. Microwave Induced Welding of Carbon Nanotube-Thermoplastic
Interfaces for Enhanced Mechanical Strength of 3D Printed Parts,
SAMPE 2015. 01-MAY-15, . : ,

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Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

<u>Received</u>   <u>Paper</u>

   TOTAL:

Number of Manuscripts:

Books

<u>Received</u>   <u>Book</u>

   TOTAL:

<u>Received</u>　　　<u>Book Chapter</u>

TOTAL:

Patents Submitted

prior to this grant:

Green, M.J., Sweeney, C.B., Saed, M.A., "Microwave-induced localized heating of CNT filled polymer composites for enhanced inter-bead diffusive bonding of fused filament fabricated parts." US Patent (pending)

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Charles Sweeney	0.98	
FTE Equivalent:	**0.98**	
Total Number:	**1**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Micah Green	0.05	No
Mohammad Saed	0.15	No
FTE Equivalent:	**0.20**	
Total Number:	**2**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Student Metrics
This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME
Total Number:

Names of personnel receiving PHDs

NAME
Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Sub Contractors (DD882)

1 a. Texas Tech University   1 b. 2500 Broadway

                                                              Lubbock   TX   79409

Sub Contractor Numbers (c):
Patent Clause Number (d-1):
Patent Date (d-2):
Work Description (e): The scope of work at TTU will focus on investigating the connection between microwave
Sub Contract Award Date (f-1): 2/1/15 12:00AM
Sub Contract Est Completion Date(f-2): 10/31/15 12:00AM

1 a. Texas Tech University   1 b. 2500 Broadway

                                                              Lubbock   TX   79409

Sub Contractor Numbers (c):
Patent Clause Number (d-1):
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Work Description (e): The scope of work at TTU will focus on investigating the connection between microwave
Sub Contract Award Date (f-1): 2/1/15 12:00AM
Sub Contract Est Completion Date(f-2): 10/31/15 12:00AM

Inventions (DD882)

Scientific Progress

See Attachment

Technology Transfer

Prior to this award, PI Green and co-PI Saed (and a graduate student) had developed IP that prompted the data that allows for this particular award. (Note: the IP was developed in its entirely prior to the award.) This award allowed us to examine fundamental scientific questions underlying the technology.

During the course of this award, our groups have been approached by a company called Essentium Materials. (Essentium Materials, LLC, was founded in June 2013 in College Station TX as a startup company primarily engaged in commercialization services for polymer composites. EM has garnered over $1MM in revenue for research services and has recently received a prestigious NSF SBIR Ph2 grant for commercialization of cellulose nanocomposites. EM is also working to develop commercial products for sale including, but not limited to, thermoplastic filament for Additive Manufacturing, 3D Printing). This company has put forward the money for the exclusive option to license the IP and is currently planning to commercialize the technology. The Green group plans to continue working with Essentium Materials (including through possible Sponsored Research Agreements).

FINAL PROJECT REPORT (DRAFT)

Title: STIR: Microwave response of carbon nanotubes in polymer nanocomposite welds

Program Officer: David M. Stepp, ARO, RDRL-ROE-M (Materials Science Division)

PI: Micah J. Green, Artie McFerrin Department of Chemical Engineering, Texas A&M University

Co-PI: Mohammad A. Saed, Department of Electrical & Computer Engineering, Texas Tech University

Dates of Grant: 2015-Feb-1 to 2015-Oct-31

Grant number: M1501433

Statement of the problem studied

Thrust 1 of the STIR project examines the heat response of polymer composites loaded with carbon nanotubes (CNTs) to microwave irradiation. This involves (1) a study of how CNT loading affects dielectric properties of polymer composites and (2) a study of how CNT loading affects the heating response to microwave radiation. Our hypothesis is that the heating of CNTs alone is not the only factor; rather, the formation of resistive (rather than capacitive) percolating CNT networks is the dominant factor in the interaction of the sample with the microwave field and the subsequent heat evolution.

Thrust 2 of the STIR project examines the effects of microwave heating of CNT-based adhesives at welds between polymer films. We hypothesize that localized CNT heating at an interface allows for polymer mobility across the interface can allow the weld to become as strong as the bulk polymer sample. We investigate such welds in both bonded polymer films and printed polymer filament structures.

For our experimental system, we choose polylactic acid (PLA) as a model polymer, given its common application in additive manufacturing.[1-3] For the nanofiller, we utilize multi-walled carbon nanotubes.

Please see original STIR proposal for more details.

Summary of the most important results from Thrust 1

Dielectric properties vs. MWCNT loading

The heating effects of microwaves on polymer nanocomposite samples are a strong function of the dielectric properties of the composites. Both DC conductivity and AC properties (AC conductivity, loss tangent, dielectric constant) were measured for MWCNT-loaded hot-pressed PLA films as a function of MWCNT loading. DC measurements were conducted using a four-point-probe. AC measurements were performed with a low-power microwave network analyzer using coaxial lines feeding a cylindrical sample holder. (Note that all of these dielectric measurements take place at very low microwave powers such that the measurement itself doesn't induce MWCNT heating.) Data in Figure 1 show AC conductivity (at 2.45 GHz) and DC conductivity, and a clear percolation transition is noted.[4-6] (Note that AC and DC conductivity values are the same for percolated, resistive networks at high MWCNT loading, but AC conductivity is higher at low MWCNT loading where capacitive effects are important.) These measurements were all taken at room temperature. Figures 2 and 3 show ε' and the loss tangent ($\varepsilon''/\varepsilon'$) as a

: 1

function of MWCNT loading. The relationship between these dielectric properties and the actual microwave heating response will be explored in further detail below.
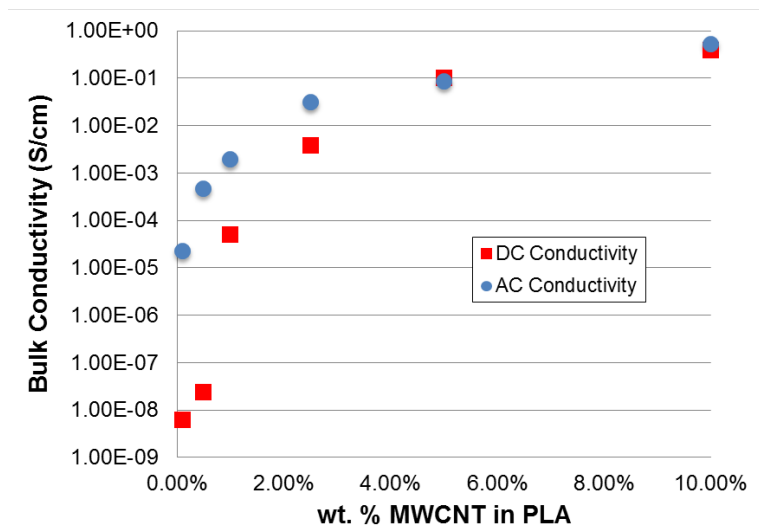

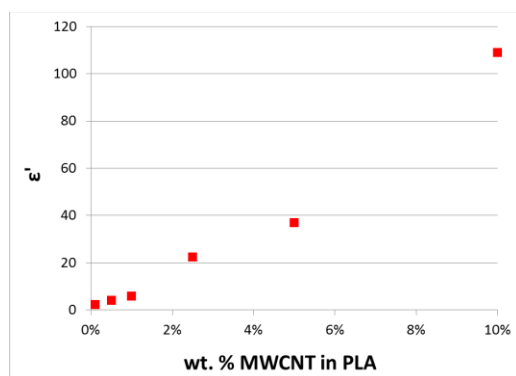**Fig. 1: DC conductivity and AC conductivity (2.45 GHz) vs. MWCNT loading in PLA films.**

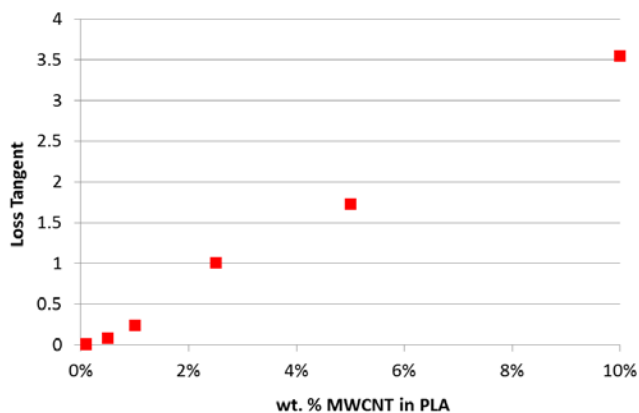**Fig. 2: ε' (real part of relative permittivity) vs. MWCNT loading in PLA films.**

Fig. 3: tan δ = ε''/ε' vs. MWCNT loading in PLA films.

Thermal response vs. MWCNT loading

We then measured the thermal response of PLA films to (10-100 W) microwave radiation at 2.45 GHz in a controlled environment. We used a forward-looking infrared (FLIR) camera to image the temperature increase of homogeneous films placed inside a rectangular waveguide (Figure 4), which in turn was connected to an Opthos microwave generator. The FLIR looks at the sample through a metal mesh window covering the open end of the waveguide; the thermal effects of the mesh itself can be subtracted from the signal through proper calibration. An electromagnetic RF meter was used to ensure safety during microwave radiation.)

Fig. 4: Custom waveguide with brass mesh window for visualization.

Figure 5 shows thermal images of PLA films at varying MWCNT loadings after 30 seconds of 20 W microwave exposure. Interestingly, the heating response dramatically increases as the loading is increased to 2 wt.% MWCNT. We hypothesize that this increase in heating response is caused by a transition from a disconnected capacitive network of the conducting MWCNTs to a connected resistor network. (One other prior study had observed a similar effect for MWCNTs dispersed in silicone oil.[7]) The induced electric current magnitudes on the MWCNTs due to microwave radiation are significantly increased above this threshold, resulting in high power dissipation through heating. We hypothesize that below this threshold, the gaps between the MWCNTs in the disconnected network inhibit current flow due to the high impedances in the matrix. Interestingly, the data also show a dramatic decrease in heating (and altered distribution) as the loading changes from 5 wt.% to 10 wt.%. From this data, we hypothesize that this is related to the transition from power absorbance to microwave power reflectance associated with high conductivity networks. The unusual, non-monotonic heating progression suggests an overall picture as follows:

- At low MWCNT loading (< percolation threshold), low microwave power absorbance is observed
- At MWCNT loading just above percolation, substantial microwave power absorbance is observed
- At high MWCNT loading, partial microwave power reflectance and lower absorbance is observed.
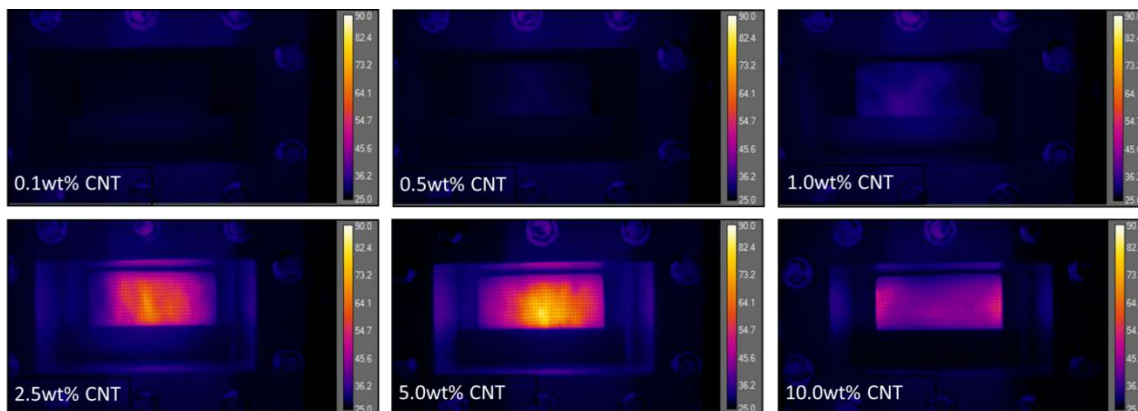
Fig. 5: Preliminary study - FLIR imaging used to capture temperature profile during microwave exposure for MWCNT/PLA nanocomposites. The non-homogeneous profile stems from both heat transfer effects and inhomogeneities in the microwave field itself.

This pattern also suggests a particular range of MWCNT loading for maximal microwave heating. In principle, such effects could be simulated in concert with heat transfer models and compared directly against these FLIR models. Follow-up studies could use finite-element modeling to undertake such a comparison. The maximum temperature from the FLIR videos are plotted vs. time in Figure 6, and the deflection from the glass transition temperature is clear.
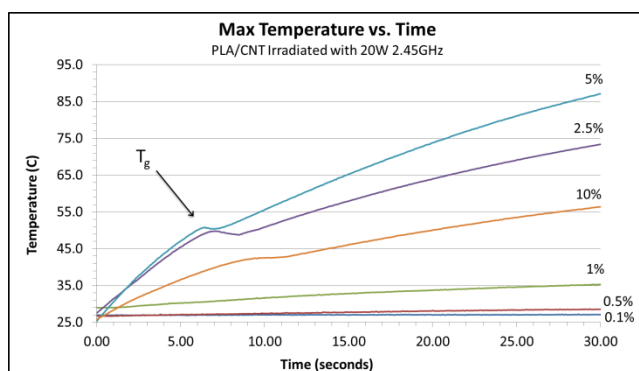

Fig. 6: Maximum temperature vs. time for FLIR videos of samples with varying MWCNT loading (microwave irradiation at 20W and 2.45 GHz).

Calorimetry

Differential scanning calorimetry (DSC) measurements were used as a point of comparison to between the thermal trace (Figure 6) with the heat capacity, melting temperature, and glass transition temperatures of the MWCNT-loaded PLA samples. Our DSC data (Figure 7) indicate that the T_g of the PLA (as measured from DSC) is correlated with a plateau (Figure 6) in the dynamic temperature vs. time response in the FLIR-imaged samples (~60 $^{\circ}$C).
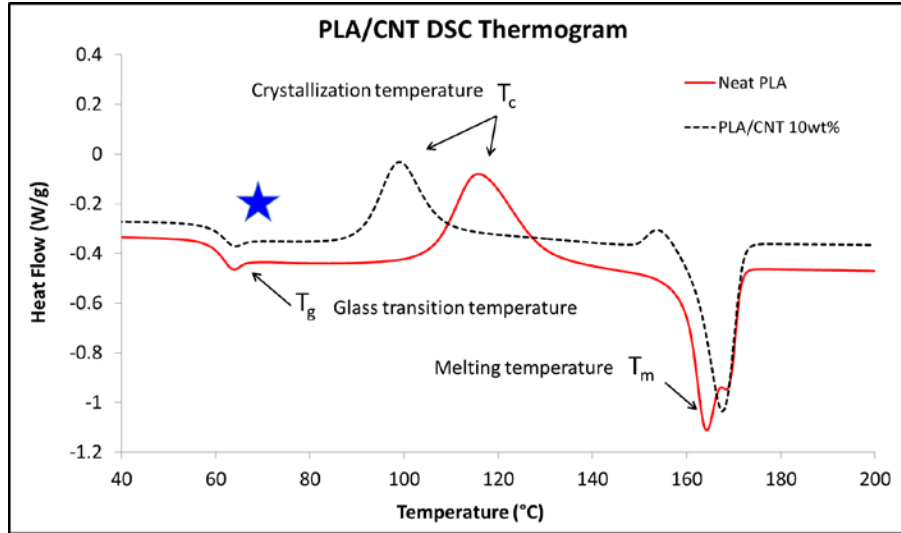
Fig. 7: DSC results for both neat PLA and 10 wt.% MWCNT-in-PLA hot pressed films. Star indicates T_g.

Heating in non-uniform materials

Ultimately, our goal is to examine not only homogeneous samples (as noted above) but also non-homogeneous samples where a MWCNT -loaded coating is applied to a bulk polymeric structure. MWCNT-coated PLA filaments were grouped into a bundle and imaged using the FLIR camera during microwave exposure (150 W) inside the waveguide (Figure 8). The localized heating at the MWCNT coating is clear from the image; we anticipate that this localized heating will manifest in increased weld strength, as investigated in detail in Thrust 2.
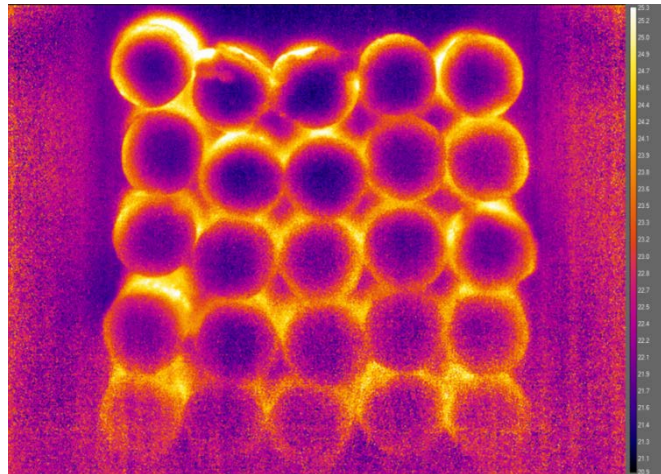

Fig. 8: FLIR imaging used to capture temperature profile during 150 W microwave exposure of a bundle of MWCNT-coated filaments

Summary of the most important results from Thrust 2

Nanotube heating at polymer film interfaces

Our primary goal in Thrust 2 was to examine direct mechanical probes of interfacial adhesion of MWCNT coatings upon microwave irradiation. In each test, a PLA film was spray-coated on a 1 in.^{2} area with a 10wt% MWCNT/PLA ink. This sprayed area was then used as an adhesive to adhere another PLA film. The MWCNT-coated area was exposed to 1250W, 2.45 GHz microwave irradiation for varying times in a custom Faraday cage/microwave setup (Figure 9).

Fig. 9: Custom microwave/Faraday cage setup for large-scale microwave exposure.

ASTM D31-63 lap shear tests (conducted using a conventional tensile tester) were used to give a macroscale indication of the effect of microwave irradiation on the enhanced adhesive strength in a between the two PLA films.

7 total tests were carried out. 4 of the 7 failed outside the bond area, indicating that the weld is stronger than the polymer itself. A typical test (Figure 10) shows that microwave radiation of thin MWCNT coatings can induce sufficient polymer diffusion to cause tensile failure to occur outside the weld. The other 3 samples broke in the welded area. 1 of these samples suffered from insufficient microwave exposure and broke in the weld. 2 of these samples suffered from overexposure and damage in the microwave. The stress-strain diagrams for these samples are indicated in Figure 11 below.
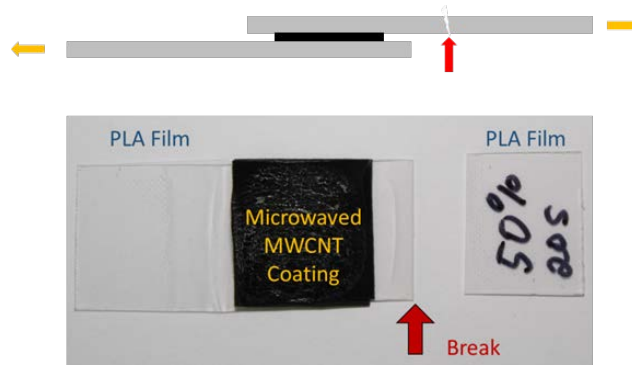
Fig. 10: Results from tensile tests of PLA films with microwaved MWCNT welds (10wt% MWCNT/PLA ink spray coated onto PLA film, bond area ~1"x1", microwaved in 1250W in a chamber at 50% duty for 20 sec)
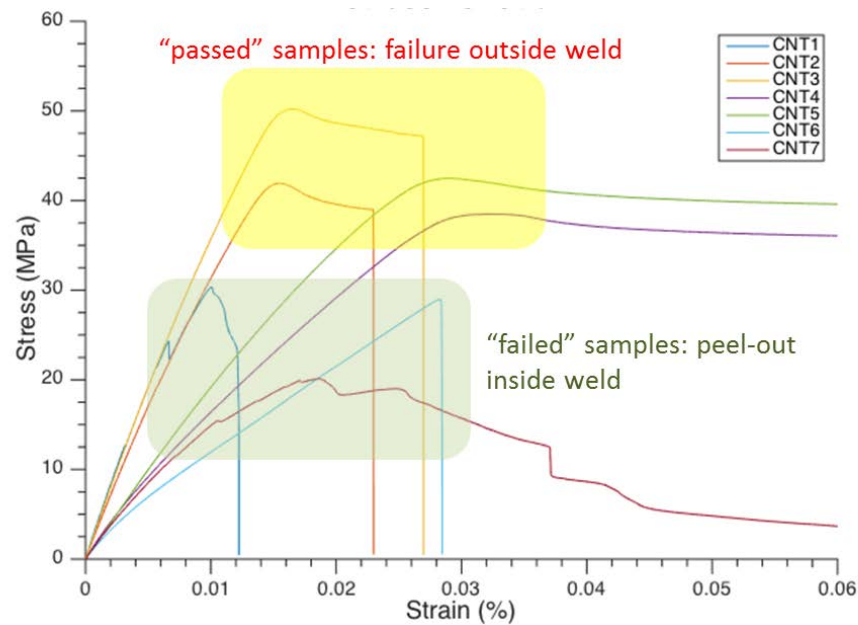

Fig. 11: Stress-strain data for lap shear tests for multiple MWCNT-bonded PLA film samples. Note the distinction between passed and failed samples; this is due to the degree of microwave exposure. Legend merely designates sample name.

(Note that the mechanical strength and toughness of the "good weld" samples are still less than what one would see in a PLA film tensile test; this is merely a typical consequence of the difference between ASTM tests since lap shear tests involve stress-concentrators near welding regions.)

These results indicate two things: (1) Given sufficient microwave irradiation, MWCNT-PLA adhesive coatings can result in excellent welds that are stronger than the surrounding polymer. With no microwave irradiation, the bonded film fails at the weld, whereas after sufficient microwave irradiation, the bonded film fails in the bulk polymer.[48,49] Additional tests are needed to narrow the parameter space (microwave power and time) where this transition occurs; thermal imaging can ensure a degree of control over this process. (2) Variation in microwave exposure can lead to variability in the heat response, which is

determined by the experimental microwave exposure chamber and ability to monitor temperature during exposure. We explore this second issue in greater detail below.

Furthermore, these macroscale tests hint at the underlying polymer dynamics at the weld, where localized heating allows polymers to migrate across the interface and remove the failure point. Additional studies are needed to extract scaling laws for the time and temperature for such polymer migration to occur.

Nanotube heating at polymer filament interfaces – thermal imaging

In similar fashion, we utilized 3D printing on an UP! Mini 3D printer to vertically print a layered structure from a MWCNT-coated PLA filament. The PLA filament was coated with a 10 wt.% MWCNT/PLA ink prior to the 3D printing process. FLIR imaging was then used to visualize the temperature field in the structure during microwave irradiation. In particular, Figure 12 shows the striated thermal response corresponding to the printed layers; localized heating effects along the welds can be seen, confirming that the heating is restricted to the MWCNT-enriched regions, promoting inter-filament welds.
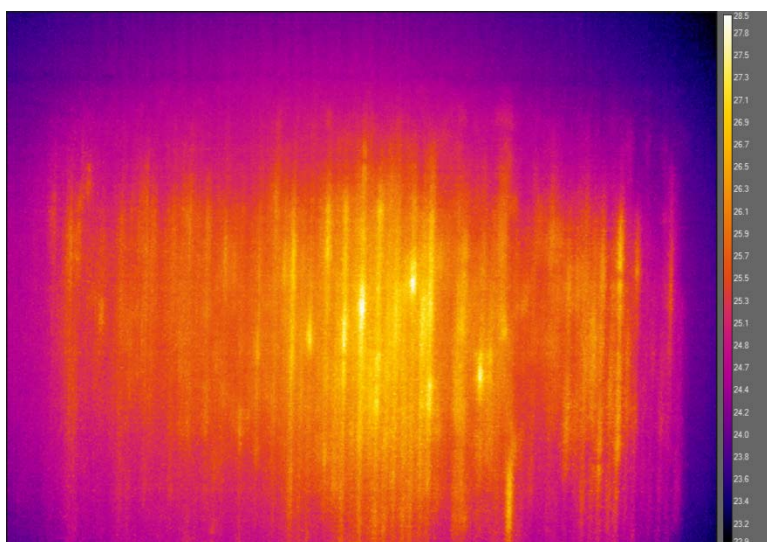

Fig. 12: FLIR image of a layered, printed structure (150 W in waveguide, 50 ms exposure)

Nanotube heating at polymer filament interfaces - mechanical data:

As an additional probe of improved weld strength, we exposed the 3D-printed structures to microwave fields using two different methods. Dogbone shapes were then bored out from these structures, and we conducted ASTM D638 tensile tests on these dogbones.

Representative tensile data from the prior ARO STIR grant is shown in Figure 14. This data was generated using the MWCNT coating, 3D printing, microwave-exposing technology described above. The vertically-printed dogbones were tested using an Instron tensile testing apparatus. The stress-strain behavior for a microwaved dogbone is shown in Figure 11 (with an exposure time of 120 seconds at a maximum temperature of 200 $^{\circ}$C) alongside stress-strain data from the baseline 3D-printed dogbone and a

bulk PLA dogbone. This data is highly repeatable because of the feedback control over time and maximum temperature in our setup.
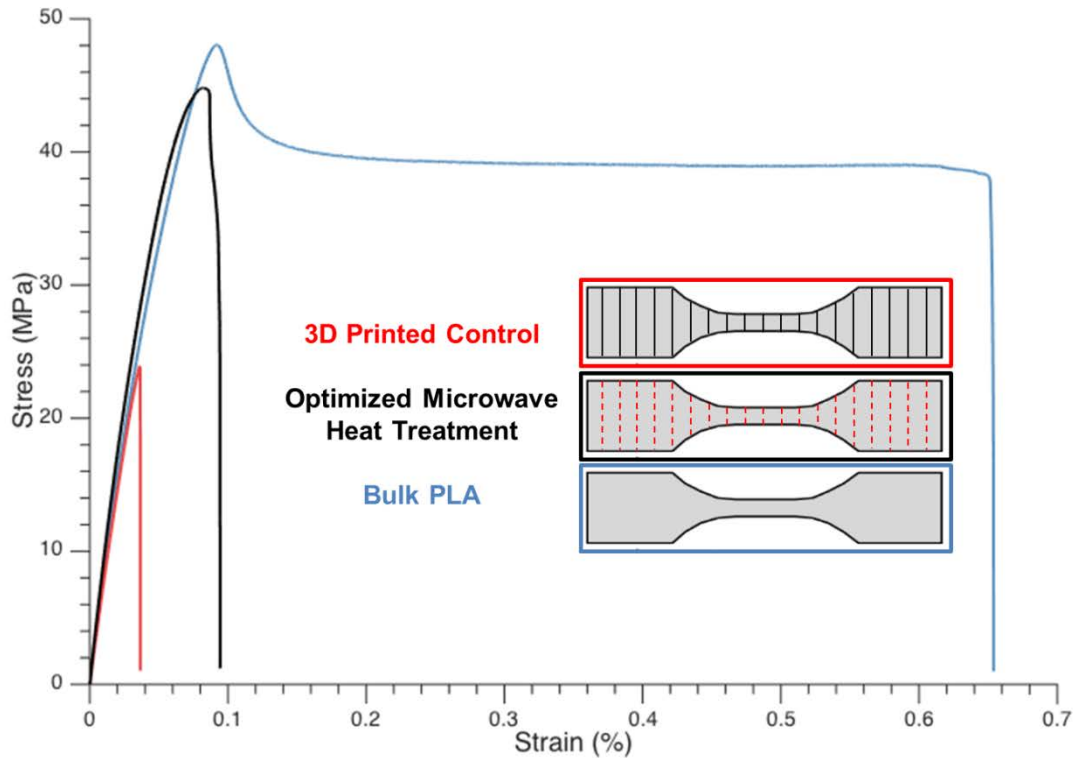

Fig. 13: Stress vs. strain for vertically-printed dogbone samples; three samples are shown: native 3D-printed samples, 3D-printed samples with microwave exposure, and bulk PLA.

There are several critical noteworthy conclusions to draw from this data in Figure 13. The use of MWCNTs at the interface allows for high localized temperatures and localized polymer mobility, such that the strength of these welds is >90% that of the bulk polymer. *This recovery in the weld strength of 3D-printed parts is unprecedented.* However, it is even more interesting to note that although the weld strength was almost completely restored, the toughness is not restored. The failure mechanism still involves a brittle failure at the weld rather than the crazing that is characteristic of the bulk polymer.

This data shows that improved weld strength in large scale systems is possible, if the microwave exposure can be controlled so that the polymer reaches temperatures sufficient to induce melting but low enough to avoid degradation.

*Solid-state microwave source / waveguide exposure***:**

In order to control the actual temperatures in the sample (rather than merely the input microwave power), the 3D-printed dogbone samples were placed in the waveguide (from Figure 4) powered by the solid-state Opthos microwave source; this source has 1W precision but only a maximum power of 150 W. Within this setup, the thermal dynamics of the sample can be monitored during microwave exposure by using the FLIR camera. The power was controlled by hand to ensure a consistent thermal history from sample to

sample (150 W up to 195 $^\circ$C, 40-50 W to keep the sample within 190-200 $^\circ$C). This means of simultaneously microwaving samples with a feedback loop based on maximum temperature allowed us to generate excellent repeatability in tensile test mechanical performance (Figure 14). The as-measured mechanical strength, toughness, and strain-to-failure are below those of Figure 13 (due to the low power on the solid-state microwave source), but these results do indicate that **a temperature-microwave power feedback loop can lead to repeatable polymer migration and weld heating on complex, macroscopic structures.**
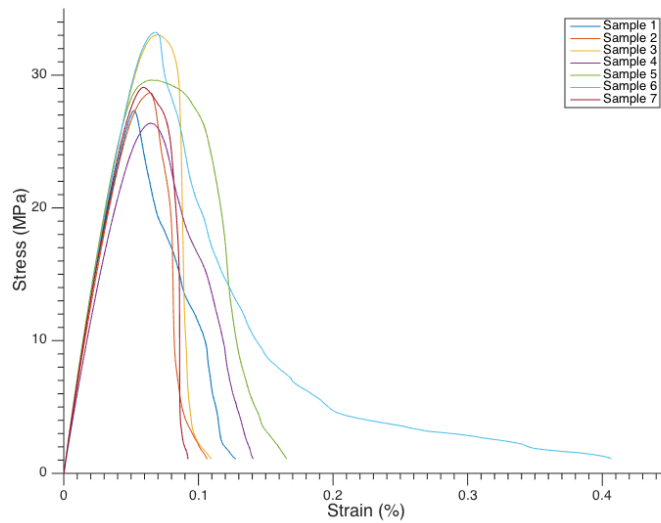

Fig. 14: Stress vs. strain for waveguide-exposed, 3D-printed samples.

Conclusions & Future Work

From this data, we conclude the following:

1. For homogeneous nanocomposite structures, our unusual, non-monotonic heating progression suggests an overall picture of microwave power transmission (in samples with MWCNT loadings below percolation), microwave power absorbance (samples with MWCNT loadings just above percolation), and microwave power reflectance (high MWCNT loadings).
2. Polymer migration across welds using MWCNT-based adhesives and microwaves allows for strengthened welds that will not act as weak spots during tension; microwave exposure (i.e., sufficient time and temperature at the interface) as the dominant factor in the pass/fail ASTM lap shear test.
3. Similarly, polymer migration across filaments in welds in 3D-printed structure allows for remarkable changes in stress-strain behavior; however, uniform microwave exposure with real-time thermal monitoring is needed to ensure repeatability and control.

These results are promising but do prompt serious fundamental scientific questions underlying many of these macroscale observations:

1. How does microwave frequency and temperature affect the dielectric properties? The dielectric data measured above is limited to the "linear" region which doesn't apply to samples in the

temperature region where welding occurs. Similarly, other key material properties such as the thermal conductivity of the nanocomposite would also change with temperature.

2. Can simulations of the coupled microwave field, dielectric response, and heat transfer dynamics allow us to make quantitative predictions for the response of MWCNT-loaded materials to microwave fields? For instance, can such simulations predict power dissipation as a function of ε' and $\tan \delta$ (which are a function of MWCNT loading, temperature, and frequency)?

3. How do time, temperature, and molecular weight affect the migration of polymers at heated MWCNT interfaces during microwave exposure? Can scaling laws be extracted to allow for prediction of polymer motion at the microscale?

4. Despite the preliminary data shown above, little is known about cooperative behavior and penetration depth limitations in the many-weld systems common to polymeric structures, including those created in an additive manufacturing context. How do such complex structures with non-homogeneous orientation and composition respond to applied microwave fields? How do the single-weld scaling laws for polymer motion on the microscale translate to macroscale many-weld systems?

5. How does the CNT loaded polymer affect the electromagnetic field distribution within the sample in a given exposure system, and in turn affect the heating profile?

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(2)   Lunt, J. Large-scale production, properties and commercial applications of polylactic acid polymers. *Polymer Degradation and Stability* **1998**, *59*, 145-152.

(3)   Tymrak, B. M.; Kreiger, M.; Pearce, J. M. Mechanical properties of components fabricated with open-source 3-D printers under realistic environmental conditions. *Materials & Design* **2014**, *58*, 242-246.

(4)   Sun, X.; Song, M. Highly Conductive Carbon Nanotube/Polymer Nanocomposites Achievable? *Macromolecular Theory and Simulations* **2009**, *18*, 155-161.

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(6)   Bauhofer, W.; Kovacs, J. Z. A review and analysis of electrical percolation in carbon nanotube polymer composites. *Composites Science and Technology* **2009**, *69*, 1486-1498.

(7)   Paton, K. R.; Windle, A. H. Efficient microwave energy absorption by carbon nanotubes. *Carbon* **2008**, *46*, 1935-1941.